Angulated Studies of the Iso-Kinetic Device for use in the Measurement of Solids Circulation Rate

Senior Thesis

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Abstract

Iron oxide-based chemical looping provides a beneficial process for coal conversion to address recent concerns for the environment. The process utilizes a cyclic system composed of two counter-current moving bed reactors and a fluidized bed reactor to achieve continuous operation and take advantage of the recyclable nature of the solid iron oxide particles present. To achieve proper design goals and full efficiency, it is important to be able to measure the solids circulation rate in the system. However, because of the harsh conditions present within the reactor system, traditional flow measurements are not feasible. The iso-kinetic device is a solids circulation measuring apparatus which avoids this issue. A separate column with solids flowing into the reactor can operate room temperature. This column's flow can be measured by traditional equipment to correlate to the solids circulation rate in system. Since the iso-kinetic device can operate at room temperature, the equipment does not need to come into contact with the harsh environment inside the reactors. Experiments were carried out on the iso-kinetic device to determine the effect angle of entry into the reacting vessel has on the effectiveness of the device. It was found that all angles test showed direct proportionality between the velocity in the iso-kinetic device and that in the reactor column. Further, correlating constants were calculated which can be used to approximate a solids circulation rate in the reactor from a flow in the iso-kinetic device.

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Introduction

Chemical Looping Technology

A modern focus in the power production industry has been on the environment and general improvements in process efficiency. The conversion of fossil fuels to energy comes with the production of significant quantities of carbon dioxide as a waste stream. Carbon dioxide is a green house gas where its presence in the atmosphere causes retention of solar radiation. Further, in traditional coal conversion processes, air is mixed directly into the reacting vessel as an oxidation source for fuel combustion. This dilutes the flue gas outlet carbon dioxide concentration making its separation more energy intensive. In the past, these streams have been discharged into the atmosphere; however worries regarding the impact this has on the environment has caused the creation of regulations which will impact new power production plants. Further, discussions of possible incentives to lower carbon dioxide emissions, such as carbon tax, have allowed this focus to potentially be monetarily beneficial. The carbon dioxide can also be utilized in enhanced oil recovery (EOR) techniques which increase the available extractable oil from a well.

To limit carbon dioxide emissions, efficient carbon capture technologies are necessary in modern processes. However, traditional methods of carbon capture, such as post combustion (MEA) scrubbers, are quite energy intensive thus significantly increasing the cost of electricity. A novel approach to coal conversion is currently being developed in the laboratory of Dr. Liang-Shih Fan[1] at the Ohio State University inherently separates carbon dioxide into a nearly pure stream eliminating the need for additional separation techniques such as gas-gas separation processes to achieve carbon capture.

Iron oxide-based chemical looping is one of the systems being demonstrated at Ohio State with a sub-pilot scale setup. The process utilizes metal oxide particles as oxygen carriers for indirect conversions of carbon fuels into a concentrated carbon dioxide stream while heat and hydrogen are generated for electricity and/or further chemical production. The oxygen carrier used is an iron oxide-based particle in the form of 1.5 millimeter diameter sphere. The particles cycle through oxidation states while exchanging oxygen between the metal oxide and the gaseous reactants. The average bulk oxidation state also cycles, as the particles return to same location in the reactor, so too does their oxidation state. The process can work with solid or gaseous fuel sources depending on process design. Figure 1 below shows a simplified schematic of the process.

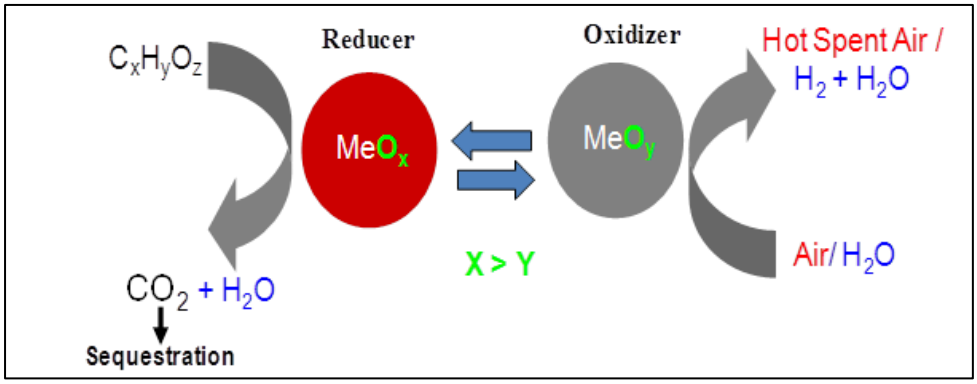

Figure 1: Conversion Phenomena in Iron-Based Chemical Looping

A carbonaceous fuel source is fed into a moving bed reactor, called the reducer, counter-currently with Fe_2O_3 iron oxide particles. The fuel source is oxidized producing a stream of carbon dioxide and steam. The steam can easily be condensed out giving a high purity carbon dioxide stream.

The reduced iron oxide particles are then fed into another counter-current moving bed reactor called the oxidizer. Here the particles react with an inlet stream of steam to produce an

outlet of hydrogen and steam. Again, the steam can easily be condensed to form a high purity stream of hydrogen. Hydrogen production is another benefit of the process adding flexibility that does not exist in traditional processes. The hydrogen can be used for fuel cells, hydrocracking, or for synthetic purposes. The outlet particles are then sent into the combustor section which operates as a fluidized/entrained bed. This section fully oxidizes the particles back Fe_2O_3 producing heat which is stored in the fluidizing gas (air) to be used to pre-heat gas streams or to generate electricity. The combustor also conveys the particles back to the top of the system allowing the reducer and oxidizer to operate under gravity, causing the looping nature of the process.

Fluidization occurs when a gas is introduced at the bottom of a packed bed of solids at a sufficiently high velocity such that the solids in the bed can be perturbed or even entrained with the gas. There are several regimes associated with gas-solid fluidization including particle fluidization, bubbling, and slugging which are examples of dense phase fluidization, and fast fluidization and dilute transport regime which are examples of lean phase fluidization. This is demonstrated in the Figure 2 below.

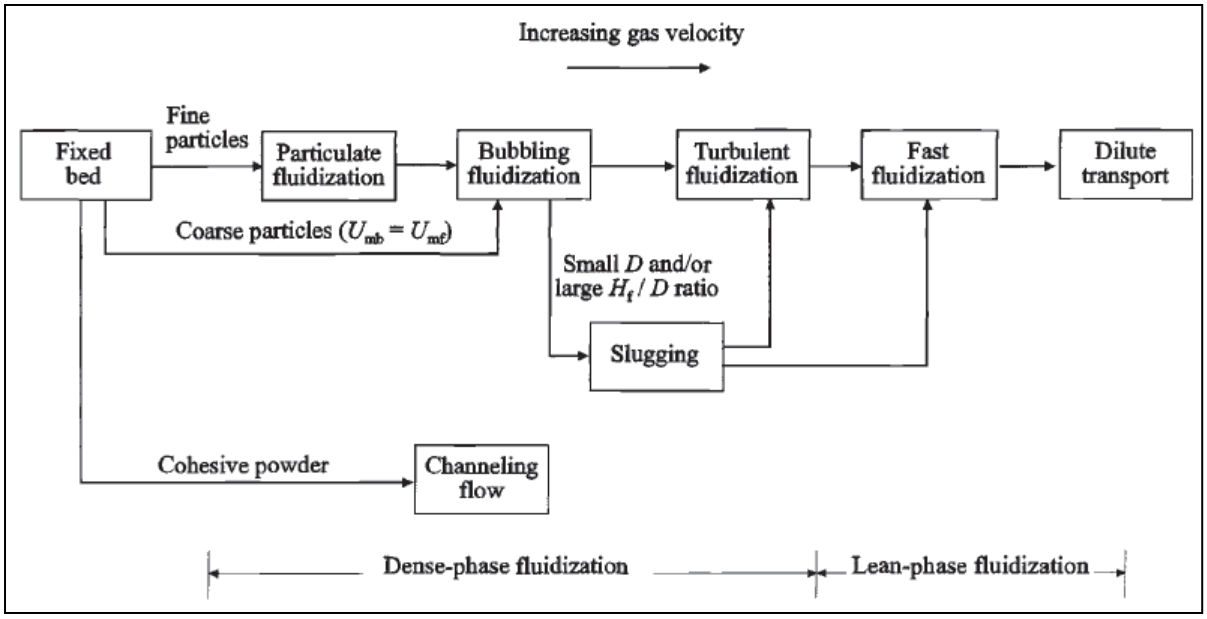

Figure 2: Fluidization Regimes with Inlet Gas Velocity[2]

The flow regimes depend on gas velocity, particle geometry and solids circulation rate making the control and measurement of these parameters important to optimum process operation.

Circulating Fluidized Beds

The OSU chemical looping process operates similarly to a CFB. In general, a CFB is composed of a riser, a downcomer, gas-solid separator and a solids flow control device[2]. The riser is utilizes fluidization, allowing the particles to move out the top, and cycle back into the downcomer which is typically a packed bed. The gas is separated from the solids at the top of the riser. This is often achieved using a cyclone. The solids then move through the downcomer, which may have a very large residence time and hold a large quantity of particles. In this way, the size of the downcomer and its outlet can be manipulated to control the rate at which solids are fed into the riser, and thus the rate of solids circulation. The solids inventory in the

downcomer also can be utilized to control solids flow. Figure 3 below shows four configurations of a CFB system.

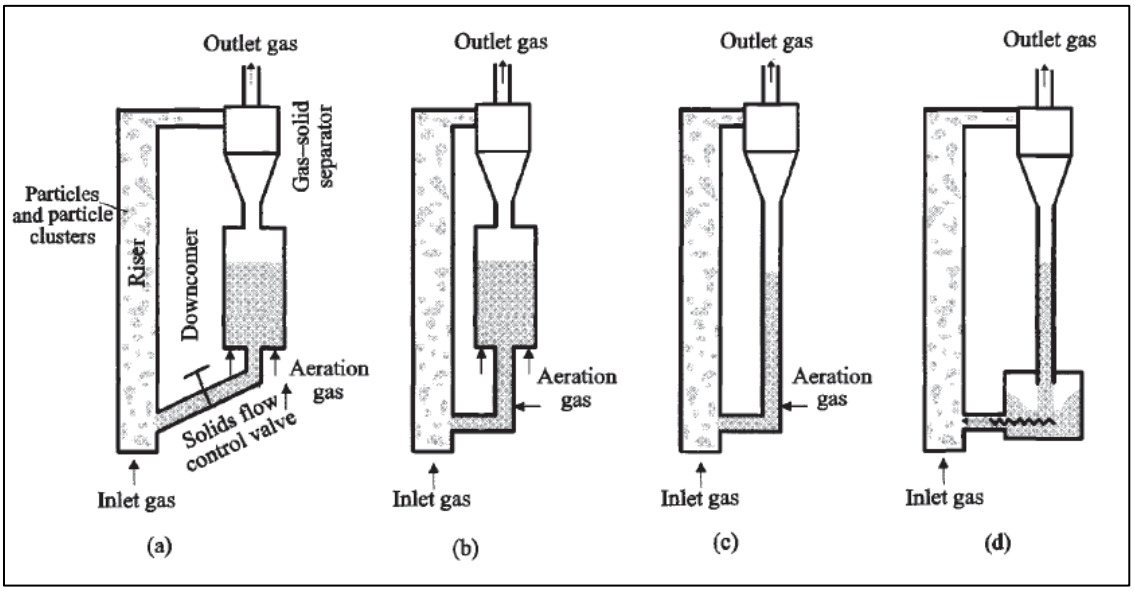

Figure 3: Four CFB Setups[2]

In a CFB, the solids circulation rate is an important design parameter when trying to meet process goals. Measurement of this quantity is more complicated than other flow measurements due to the complicated nature of solid-gas flow. Irregular velocity profiles, particle size distributions, particle accumulation at the column walls, and even moisture content affect the accuracy of measuring solids circulation rate[3]. Its reliable measurement has been an issue that has needed a solution[4]. For example, a desired power production capability is associated with a required amount of fuel. The amount of fuel corresponds to an amount of oxygen associated with the iron oxide oxygen carriers. Thus, to optimize the process it is important to be capable of measuring and controlling the solids circulation rate. Also, the type of flow regime that occurs in the riser section is a function of solids circulation rate for lean phase fluidization. In a reacting

system, controlling the flow regime in the riser is necessary to meeting efficiency goals. Thus it is important to be able to accurately measure the solids circulation rate.

If the CFB columns are fabricated out of a material that is transparent, such as glass or acrylic, then the motion of the bed could be used to determine a solids circulation rate. Or a window could be used in a location so that the entire apparatus need not be transparent. However, many processes, particularly ones with a chemical reaction occurring, operate at high temperatures and pressures. In the iron oxide-based chemical looping process, the reducer operates at 900^{o}C, the oxidizer at 850^{o}C, and the combustor at 1100^{o}C. This limits the range of feasible reactor construction materials typically to metals, ceramics, or some combination thereof. Further, in commercial scale systems operating at high temperature, the walls of the reactor are refractory lined for thermal insulation. These thick walls make windows or measurement probes that require column wall penetration infeasible.

A few devices are currently in use to measure solids circulation rate. Capacitance probes can be used to measure the quantity based on the capacitance of a column in the region of measurement. However, this technique is invasive to flow and cannot operate at high temperatures. Tuning forks measure flow based on resonance from particle bombardment, but suffer the same issues as capacitance probes. Nuclear gauges can also be used to measure solids circulation rate, and they are non-invasive and capable of being used for high temperature systems. Though they can be used in reacting systems, the technology used requires licensing and causes other issues such as requiring specialized personnel and potential health risks.

The Iso-Kinetic Device

A configuration that can be used to measure solids flow rate in CFBs operating under the extreme conditions in a reacting system is known as the Iso-Kinetic Device (IKD). The device is composed of a separate, smaller diameter column entering the packed bed region where the solids flow measurement is desired. The smaller column can be extended away from the harsh reactor environment where the solid flow measurement can be taken at room temperature, and even low pressure depending on the design. This configuration allows for the use of reliable, low-cost, nonhazardous probes. Solids are fed into the smaller column which can be opened periodically such that solids flow into the CFB. The change in bed height in the smaller column correlates to a respective change in bed height in the larger column. This change in bed height can be used with the cross sectional area of the column to calculate a volumetric flow rate. The device is no different than other solids inlets into the column and can be considered minimally invasive to the system. Figure 4 below shows a schematic of an IKD attached to the downcomer of a circulating fluidized bed.

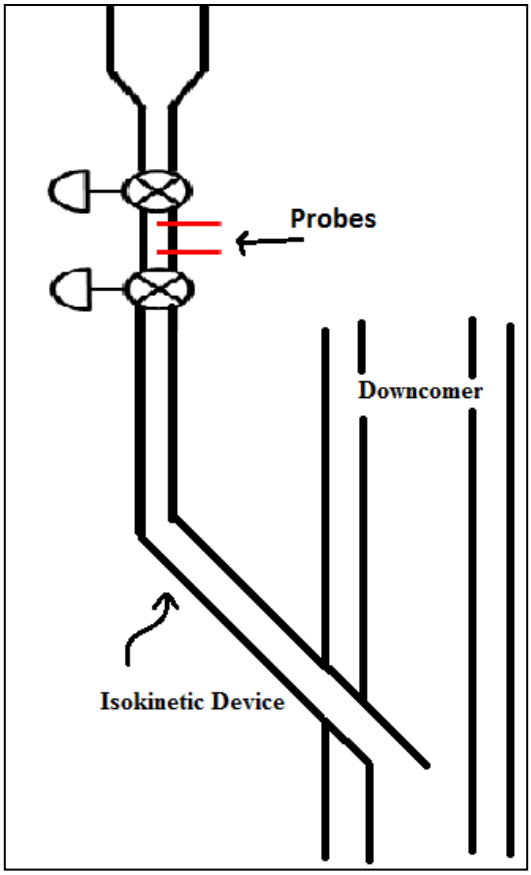

Figure 4: A Schematic of an Iso-Kinetic Device

This setup is quite similar to the iso-kinetic device that would be utilized on the pilot scale iron oxide based chemical looping system currently being developed. Solids can be charged into the hopper at the top of the device. When a measurement is desired, the valves can be opened for a given period. The time required to the bed height to move passed the top probe to the bottom probe could be used with the distance between them to find a bed velocity. This velocity could then be correlated to a respective bed velocity in the downcomer.

Literature Review

A literature review was conducted to gather information regarding circulating fluidized beds, processes that utilize CFBs, and other topics relevant to this research. It was also desired to find literature discussing issues with flow rate measurements in CFBs. Some of the relevant external information found will be discussed in this section below.

Fan *et al*[2] provides an extensive discussion of gas-solid flows, including the underlying principles such as particle collisions and heat transfer, as well as specifics on devices and equipment involved in systems utilizing gas-solid flows. A particularly useful section of the book for the purposes of this research was a discussion of CFBs. The general setup is described along with different configurations. Figure 5 below is taken from the book.

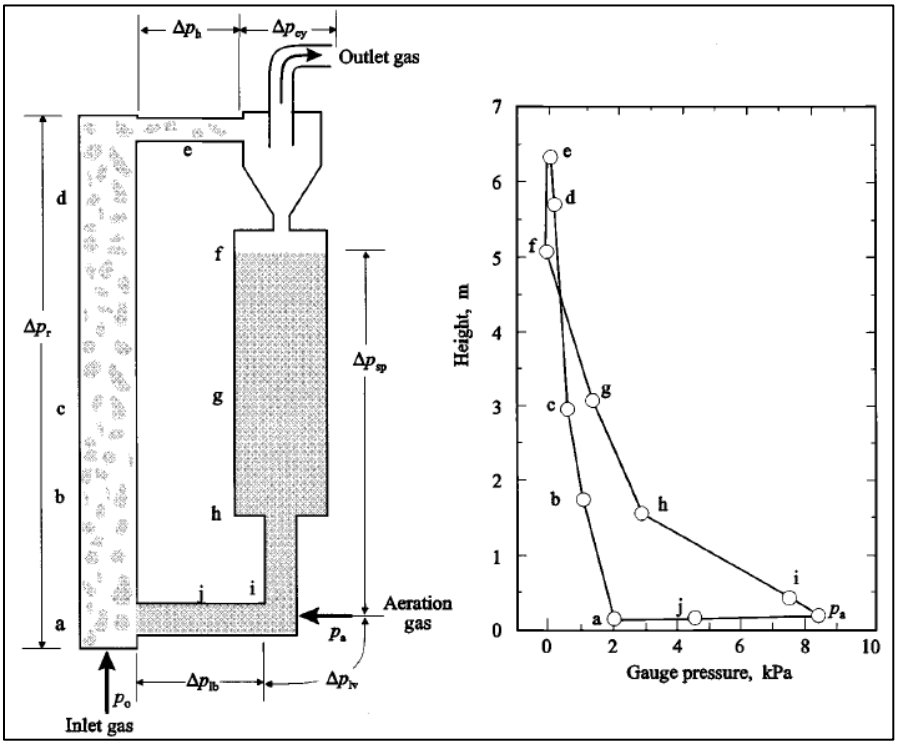

Figure 5: (a) A Circulating Fluidized Bed (b) Height vs Gauge Pressure Around a CFB[2]

The figure shows the differences in solids hold up between the riser section, where the solids are being fluidized, and the downcomer where the solids are being conveyed by gravity in a moving packed bed configuration. The figure also demonstrates the several pressure differences associated with a circulating fluidized bed. The pressure differences are marked along with specific locations on the CFB and the graph shows the gauge pressures at these locations. Depending on reactor section, the pressure differences have a non-linear dependence on height as the particles are conveyed around the system. The pressure drop in the riser is associated with gravitational energy requirements necessary to convey the particles. The pressure drop in the downcomer is associated with pressure built up from the height of the moving bed from gravity. It is important to ensuring smooth operation of a CFB that pressure is regulated so that no imbalances occur.

Fan gives methods of calculating or approximating these pressure drops based on system parameters such as the gas flow rate, particle density, and particle type. These approximations can used in correlations to roughly predict solids circulation rate. However, the correlations depend on a large number of properties of the system and the particles limiting the reliability of these techniques.

The nature of solids flow in the riser of a circulating fluidized bed depends on particle type, gas velocity, and solids circulation rate. The dependence of solids holdup in the riser section on solids circulation rate can be seen in Figure 6 below.

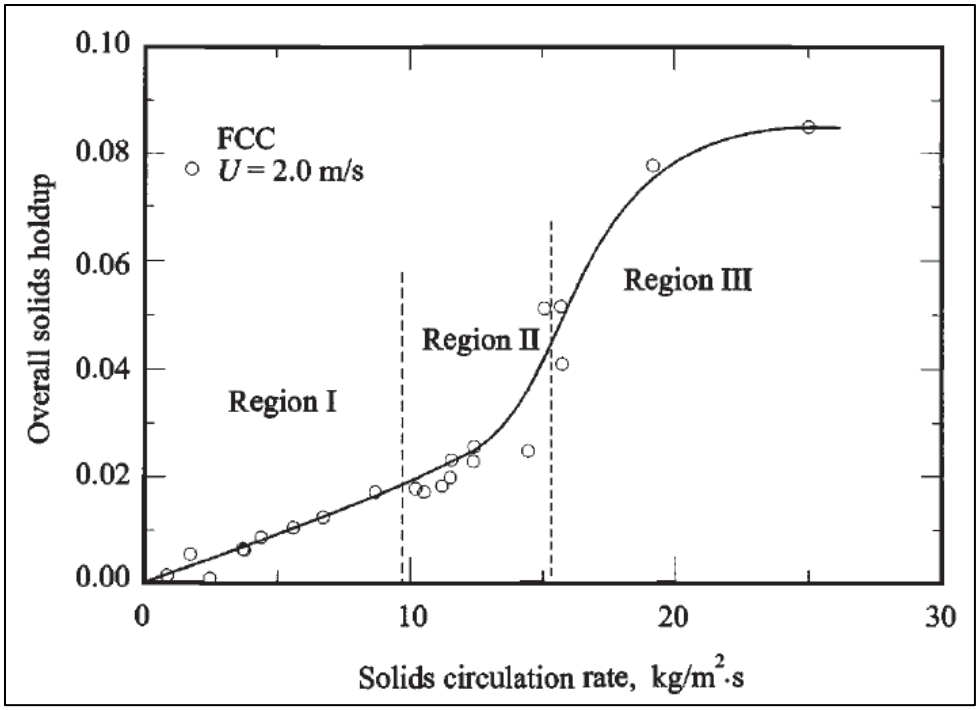

Figure 6: Overall Solids Hold Up vs Solids Circulation Rate[2]

The first region is associated with a linear dependence of solids holdup in the riser on the solids circulation rate. The second region shows a rapid slope increase, which is explained in the text as being caused by reflux of the solids at the top of the riser. Finally, region III is associated with "fast fluidization" and shows a drop-off in the rate at which the solids holdup increases with solids circulation rate. The importance of solids circulation rate on the flow regimes in the riser makes reliable solids flow measurements important to the proper design, safety, and optimization of a circulating fluidized bed, and this is part of the motivation for this research.

Mattisson *et al*[5] proposed a process for the conversion of carbon based fuels with the separation of carbon dioxide inherent in the process. Thus, the process is environmentally friendly which is becoming more important as focus on sustainability and emissions reduction

increases. Iron oxide particles are used as oxygen carriers in the chemical-looping combustion process. A simplified schematic of the process is shown in Figure 7 below.

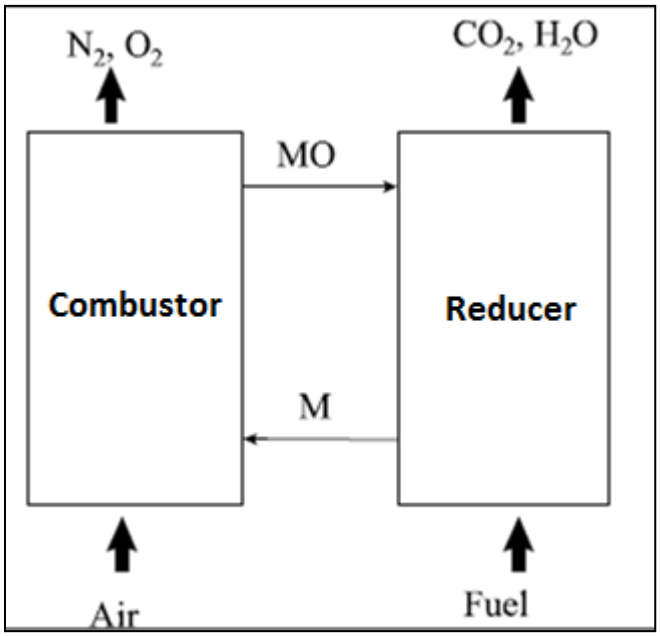

Figure 7: A Basic Schematic of a Chemical Looping Process[5]

The system is composed of two interconnected fluidized bed reactors: the combustor and the reducer. The combustor is a riser utilizing a high velocity fluidized bed which oxidizes the particles and conveys them into the reducer which is a low velocity bubbling fluidized bed. The reducer is fed with high oxidation state metal oxide particles which move counter-currently to a fuel feed. The particles are reduced, and the fuel is converted to a stream containing carbon dioxide and steam. The steam can easily be condensed producing a high purity stream of carbon dioxide which does not require further purification. Thus, carbon capture is inherent to the process giving it an edge over traditional carbon –based fuel conversion processes.

A similar iron-oxide based chemical looping process is currently being developed by Fan[1]. Two sub pilot scale processes are being investigated on campus at the Ohio State

University. One utilizes coal syngas, while the other processes powdered coal solids directly. Figure 8 below shows a schematic of the syngas process.

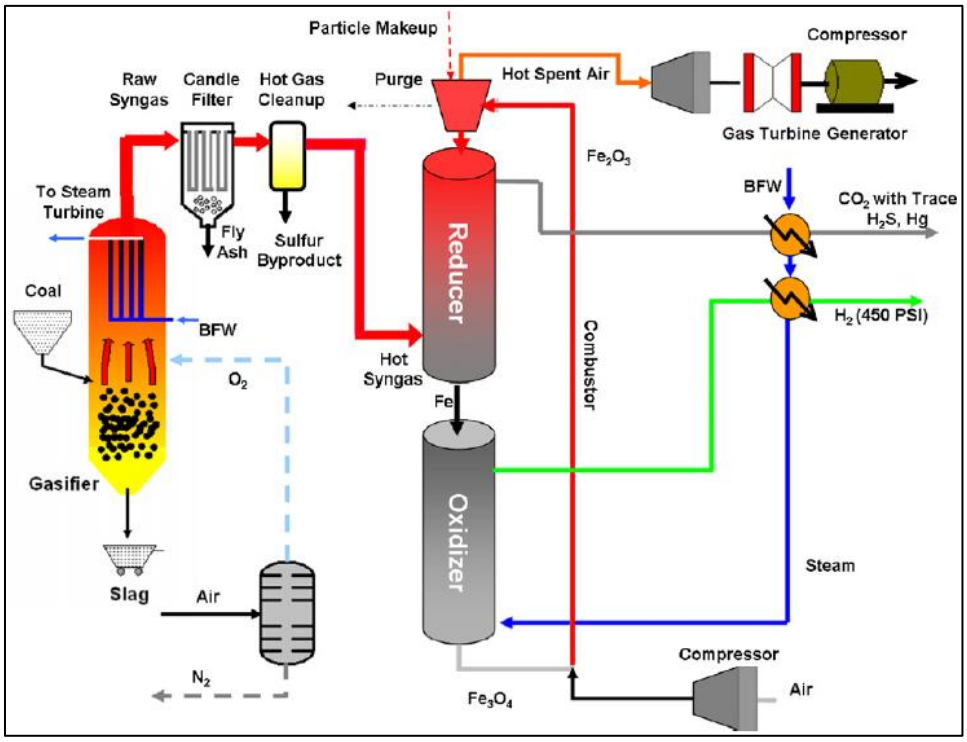

Figure 8: A Process Diagram of the Iron Looping Process[1]

The coal is first converted into syngas with a gasifier. Sulfur and particulate matter in the raw syngas are cleansed from the stream. The clean syngas then enters the reducer.

The reducer is a moving bed reactor that is counter-currently fed with high oxidation state iron-oxide particles from above and the carbon fuel source from below. The reducer converts these components into a stream of carbon dioxide mixed with steam and reduced iron oxide particles. The steam can be condensed out, producing a high purity stream of carbon dioxide which does not require further purification, thus, the energy requirements associated with carbon removal is minimized.

The reduced particles then move into the oxidizer, which also a moving bed reactor. The countercurrent feed to the oxidizer is the outlet particles from the reducer from above, and steam near the bottom. This reactor produces a stream of hydrogen mixed with steam and the partially oxidized iron oxide particles at the bottom. Similarly to the reducer, the steam can be condensed out producing a high purity stream of hydrogen. The hydrogen can be utilized in a variety of ways including but not limited to energy generation from fuel cells, hydrocracking for purification of hydrocarbons, or even synthetic purposes. The reducer and the oxidizer operate in a moving bed reactor configuration to take advantage of reaction equilibrium and thus maximize efficiency.

The final section of the process is the combustor, which is a fluidized bed reactor. The purpose of the combustor is to return the partially oxidized particles from the oxidizer to the high oxidation state of the reducer feed. Additionally, fluidization is used to convey the particles against gravity to the top of the reducer such that the moving bed reactors can operate under the effects of gravity. The reactor is fed with the outlet from the oxidizer and the fluidizing/oxidation gas: air. The oxidation in the combustor is exothermic and thus heats the air which creates a high temperature stream of air, in addition to the high oxidation state particles, which can have energy extracted from it.

The Ohio State iron looping processes utilizes a reactor setup similar to a circulating fluidized bed. The reducer-oxidizer portion of the system can be viewed as similar to a downcomer, while the combustor is similar to a riser. This iron oxide based chemical looping process is advantageous to the one previously described in that a pure stream of hydrogen can be produced. This is because the reducer is capable of taking the feed Fe_2O_3 down to Fe/FeO which allows the steam-iron reaction to be possible in the oxidizer. This makes the process more

flexible and allows full utilization of the capabilities of the iron oxide oxygen carriers. Another unique feature of the process is the counter-current moving beds used. It allows full reduction of the Fe_2O_3 particles, while still achieving full fuel conversion.

Gauthier[6] discusses challenges faced by fluidized bed systems with specific reference to feasibility in the refining industry. Solids circulation rate measurement is addressed as one of the most important design parameters for a circulating fluidized bed. Despite its importance, measuring this parameter is concluded to be quite difficult. Use of pressure drop correlations and sampling the flow along the riser are presented as two possible methods for solids circulation rate measurement, but are often associated with significant errors.

Some early uses of iso-kinetic[7] devices were for gas analysis. One patent filed in 1994 describes a device used to analyze a multi-phase stream exiting a separator for the purposes of optimizing the feed rate into the separator. Fluid samples would be taken using an apparatus capable of iso-kinetic sampling, such that the separated portions of the stream would have the same temperature and pressure as the original separator outlet stream. The device pulls two samples simultaneously through two orifices: one counter-currently and the other co-currently downstream. The streams are then sent into a container where heat can be exchanged.

The multi-phase stream may include entrained droplets of liquid or may mostly be gaseous. The purpose of the container where heat exchanging takes place is to either heat the sample such that condensate drops/particles can be evaporated fully or cool the sample such that they condense into the liquid form. The remaining gaseous or liquid bulks can then be sent to be analyzed by density measurement equipment, or gas chromatography/infrared photo-spectroscopy. The measurements taken on the samples can be used to optimize feed rate into the

separator, which is having its outlet analyzed, such that efficiency is maximized. A schematic of the device is shown in Figure 9 below.

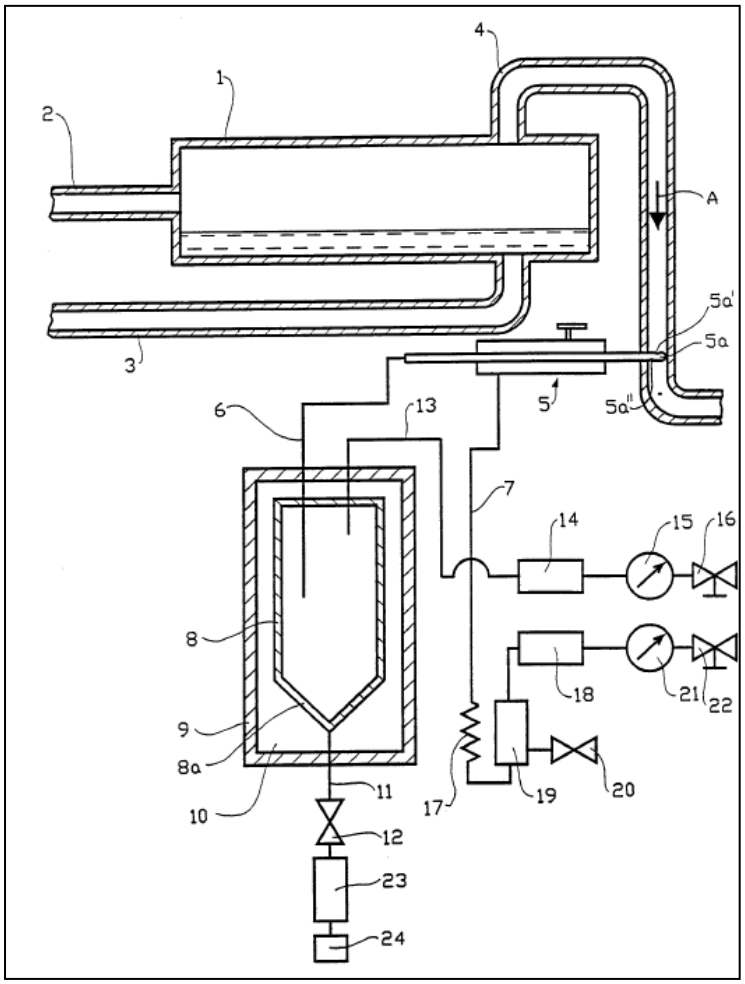

Figure 9: A Schematic of an Iso-Kinetic Multi-Component Fluid Analysis Device [7]

The outlet from a separator (1) is sent through the iso-kinetic apparatus (5) via the two orifices for counter-current (5a') and co-current (5a") sampling. The counter-current sample is piped (6) into the container (8) where heat exchange can take place. Liquid can exit out the bottom (11), and gaseous components may exit the top (13). Streams 7, 11, and 13 can then be

sent through analysis equipment such that the quantity of entrained liquid in the outlet of the separator can be determined and thus the feed into it can be optimized.

Experimental Methods

The Reactor

The apparatus used for the experiments was a cold model reactor fabricated out of acrylic with a height of about ten feet. The reactor was composed of the three inch column, the one inch column, a section for each of the ten angles with an attachment for the one inch column and the outlet at the bottom fitted with a valve to control particle flow. All of the parts except for the one inch column were attached to each other by hand tightened nuts and bolts and stabilized to a rigged frame with a round clamp. The middle sections included a short, one inch diameter column projecting from the side at a specific angle. There was an expanded section for each of the angles (in degrees): 30, 35, 40, 45, 50, 55, 60, 65, 70 and 80. The one inch column included a soft plastic tubing portion which could be fitted into the attachment on the expanded section to match its angle and a platform at the top of this column was secured to one atop the three inch column using a clamp. The one inch and three inch columns had strips of paper with inch markers taped to the sides so that proper measurements of bed height changes could be made for both columns. Figure 10 shows a schematic of the experimental apparatus.

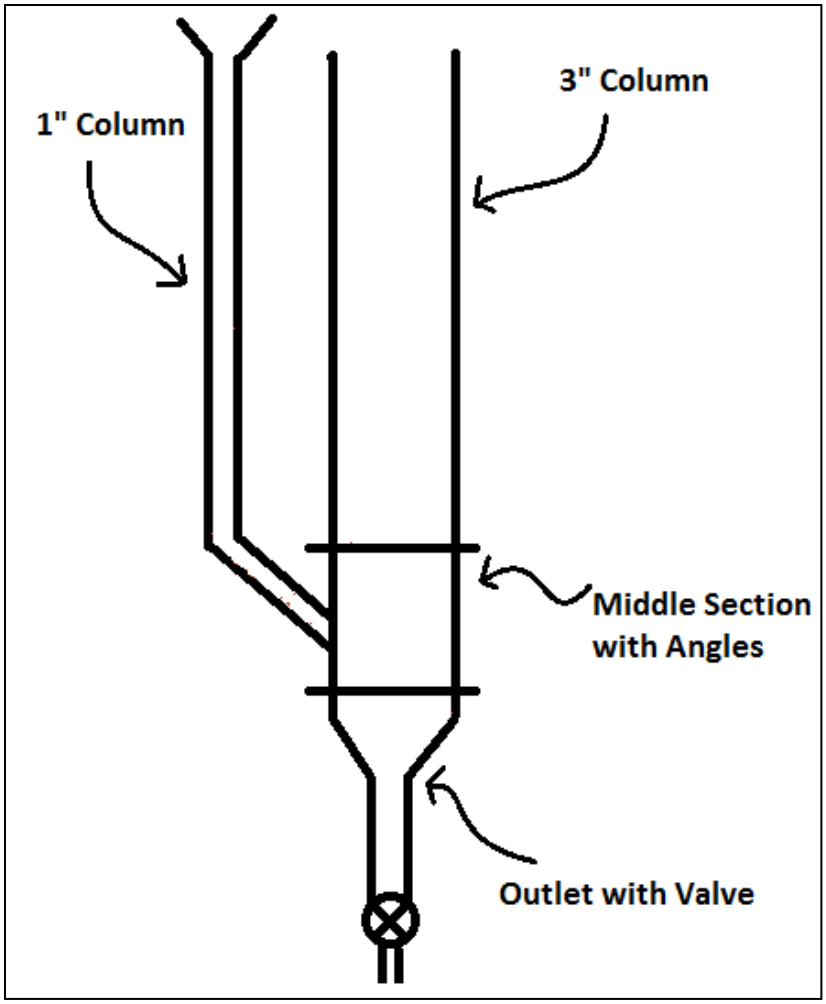

Figure 10: The Experimental Apparatus

Experimental Procedure

The apparatus described was assembled for an expanded section with one of the angles. The entire reactor was filled with 1.5 mm iron oxide particles such that both one inch and three inch columns were filled above the top of the measuring paper. A point was marked a few inches down on the paper of the one inch column to denote an initial bed height for the measurements. The top of the paper was not selected for this to ensure steady state flow development in the columns before data collection. A point 10-14 inches below the starting mark was selected on the

one inch column to serve as a termination point. A stopwatch was used to keep track of time, so bed velocity could be established. The valve at the outlet of the reactor was opened so that particle flow began. Once the top of the bed in the one inch column reached the initial marking, the stopwatch was started and the location of the top of the three inch column bed was noted. Once the one inch bed reached the termination point, the timer was stopped, and the valve was closed. The process described was considered one run and bed height changes and time were recorded as the data. The particles that exited the column during the run were collected at the bottom in a bucket and recharged into the top of the reactor to refill to above the measuring paper in each column. At this point, another run was performed to account for differences in percent valve opening and other possible errors. Four or five runs were performed for each angle. Once all runs were done for a given angle, the column was emptied to just below the middle section so that the reactor could be disassembled at this point, and a new angle could be tested. This process was repeated for each of the ten angles.

Data Analysis

Notation		
Symbols	Units	Name
Vi	cm^3/s	Volumetric flow rate of column i
vi	cm/s	Velocity of column i
V'i	cm^3/s	Volumetric flow rate of column I at insert
v'i	cm/s	Velocity of column I at insert
Hi	cm	Height of column i
t	s	Time of run
Di	cm	Diameter of column i
Di'	cm	Diameter of column I at insert
Theta	degrees	Insert Angle
Ai	cm^2	Area of column i
A'i	cm^2	Area of column I at insert
m	-	Correlation factor
b	cm/s	Intercept of fit

Figure 11: Notation for Data Analsys

The angles 35, 40, 45, 50, 55, 60, 65, 70, and 80 degrees were tested using the experimental apparatus previously discussed. Each angle had four or five runs performed with attempts made to vary percent valve opening. The data collected for each run was composed of the steady state change in bed height of both the three inch and one inch columns. The time was also recorded so that velocities could be calculated. The raw data can be found in the appendix.

For each run, a velocity was calculated for each column by dividing the change in bed height by the time. This is shown in the equation below.

$$vi = \frac{\Delta Hi}{t} \quad (1)$$

The velocities calculated can be used to calculate the local velocity of each of the columns right at the insert of the one inch column. Assuming bed velocity in the three inch column only varies with height and not radial or angular position and constant bed density, these local velocities should be equal for both columns. Figure 12 below shows where the calculated velocities and the insert velocities are located. The diameters with primes indicate they are associated with the insert location.

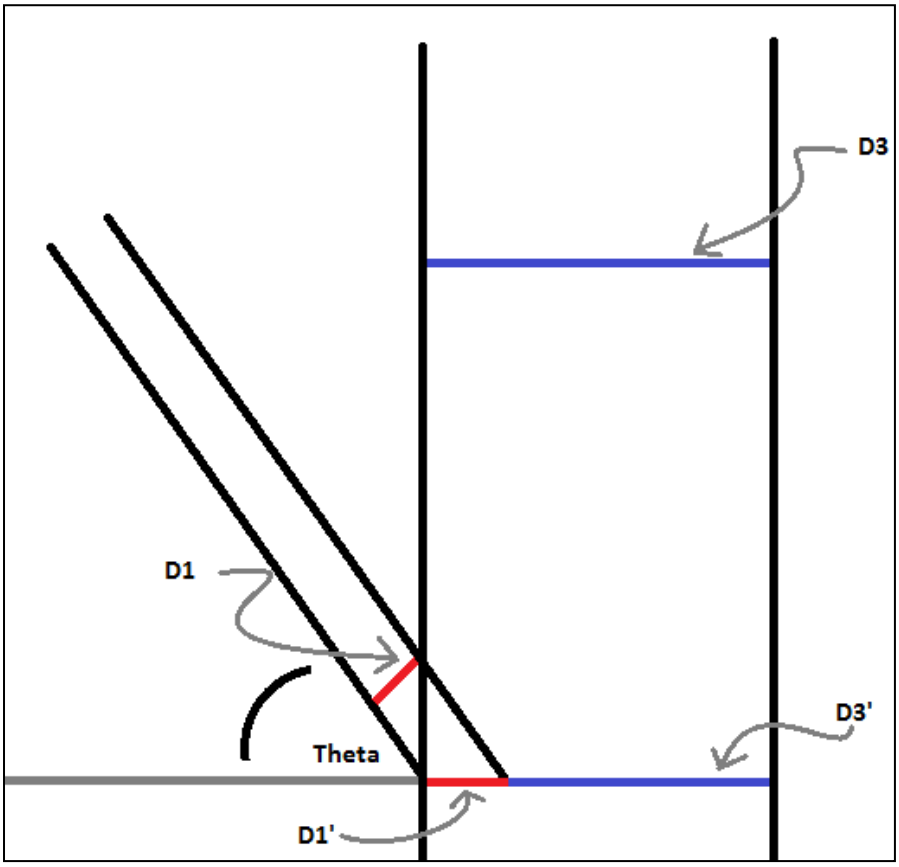

Figure 12: The Actual and Insert Location Diameters

The cross sectional area of both columns near the insert must be calculated, as it is different for both columns than the upper regions where measurements were taken. The area of

the one inch column can be calculated using trigonometric expressions and the angle of column entry. The calculations can be performed using the equations below.

$$\sin(90 - theta) = \frac{D1}{H}$$
$$H = \frac{D1}{\sin(90 - theta)} \quad (2)$$

The height of the insert can be used to find the diameter of the lipped portion of the column at the insert as follows.

$$\tan(theta) = \frac{H}{D1'}$$
$$D1' = \frac{H}{\tan(theta)} = \frac{D1}{\sin(90 - theta) \times \tan(theta)} \quad (3)$$

The area can be approximated as an oval due to the lip added to the column such that the outlet is flat. Figure 13 below shows how the calculated and measured diameters relate to the cross sectional area of the one inch column at the insert.

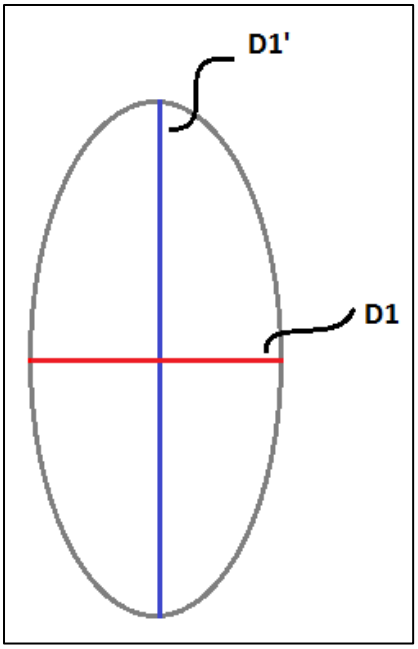

Figure 13: The Characteristic Diameter of an Oval

The area of the oval can be calculated using the two diameters as shown in the following equation.

$$A = \frac{D1}{2} \times \frac{D1'}{2} \times \pi \tag{4}$$

The velocities near the insert can be calculated using conservation of mass assuming the bed density does not change when the cross section area of flow decreases. The equation to calculate the three inch column velocity and the one inch column velocity at the input both come from a volume balance on either column. The form of this balance is shown in the equation below.

$$Vi = \text{Ai*vi=Ai'*vi'} \tag{5}$$

Results

The insert velocities can be calculated using the results of the experiments and the measured cross sectional area of the columns. The insert velocities of the three inch column were plotted against those of the one inch column for each run at a constant angle. These curves were expected to be mostly linear. This should be the case as the velocities at the insert were assumed to be equal in both columns. However, it was expected that a linear trend may not exist at certain angles, possibly those near the upper or lower extremes of angle. Figure 14 below shows the plot generated for each angle tested.

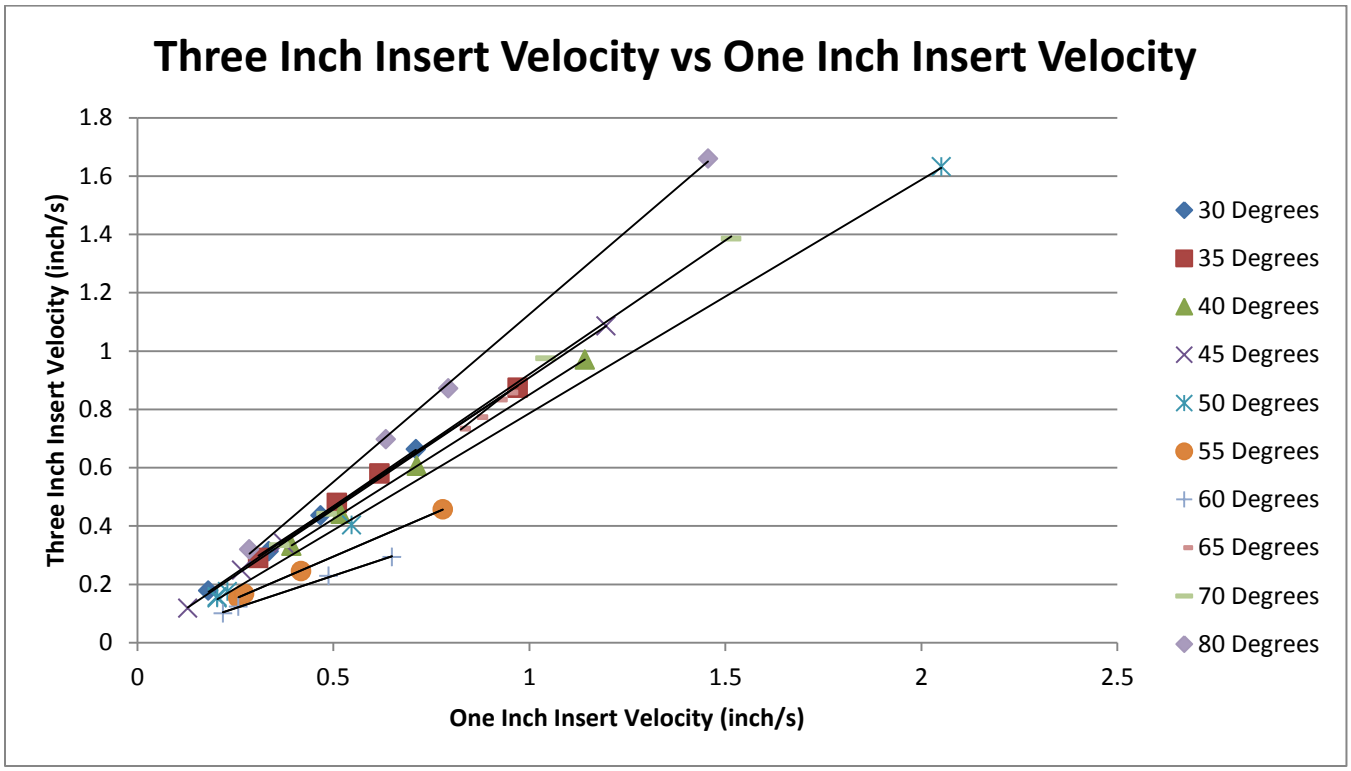

Figure 14: Three Inch Insert Velocity vs One Inch Insert Velocity for Each Angle

A linear fit performed for each angle gave great fits with R^2 values very close to unity. A linear fit performed for each angle showed near zero intercepts. This is expected since if the one

inch column is not flowing, the three inch column should not have a bed velocity. This would be the case when the valve is shut. At steady state, if the valve is shut and there is flow this would indicate a mechanical issue with the valve. The slopes of these fits are mostly very close to one. Figure 15 below displays this with the slopes of the fits as a function of angle.

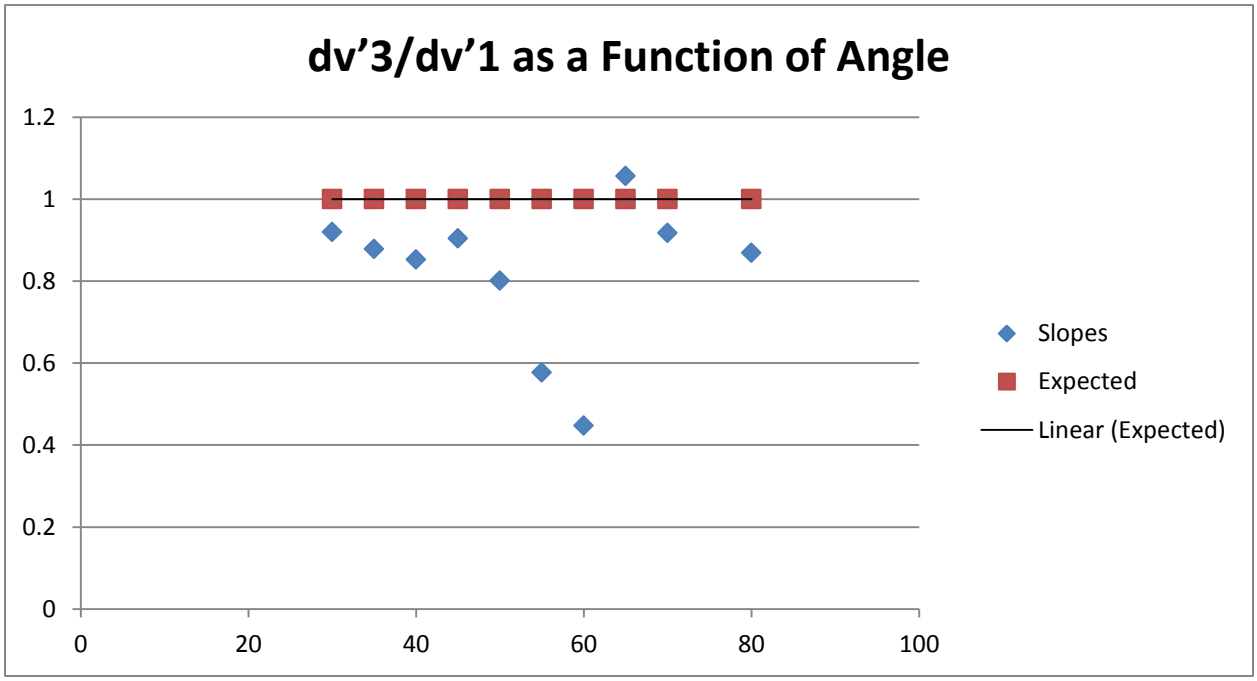

Figure 15: Slope of Linear Fits as a Function of Angle

The red dots denote the expected values. The predicted slope was unity because the velocities are assumed to be equal, and thus a change in one should correspond to an equal change in the other. The experimental data agrees with this prediction for most of the angles tested; however at angles of 50 degrees through 60 degrees, the slope becomes smaller, reaching nearly 0.4 before increasing back towards unity. However, for all of the data except for 65 degrees, the slope of the best fit lines is below one. This indicates a lower velocity in the three inch column than that in the one inch column.

Another interesting feature of the linear fits for the three inch velocity as a function of one inch velocity is how close the measured intercepts are to zero. Figure 16 shows the intercepts of the measured linear fits as a function of angle.

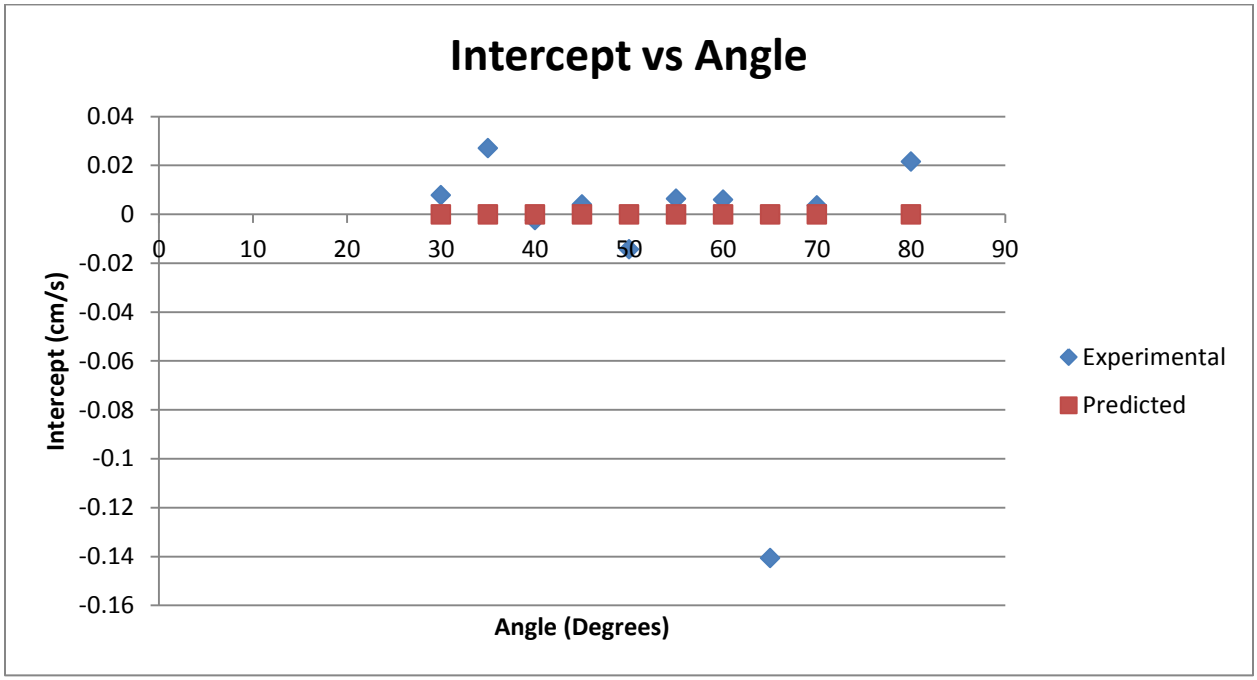

Figure 16: Intercept of Linear Fit as a Function of Angle

Almost all of the intercepts are within about 0.03 cm/s of the predicted value of zero. However, there is an outlier at 65 degrees where the intercept reaches nearly -0.14 cm/s. This predicts a negative velocity in the three inch column for a closed valve which is certainly not realistic. It is likely that the difference between all of the intercepts and zero is due to experimental error.

Sources of Error

In the experiments described above, there existed a few sources of human error. For example, because the beds in both columns were in motion, noticing that the one inch column

had reached the starting mark, starting the timer, and marking where the bed was on the three inch column took time. Somewhat more significantly was this effect at the termination of a run. The added step of closing the valve so that flow stopped added some bed height change to that which was intended. These issues could be almost completely eliminated if more than one experimenter is present, though this was not always possible.

Finally, steady state flow might not have always been achieved. This would cause any associated bed height changes to lose much meaning. It the flow is at steady state, the velocities of both beds will be constant through a run. If the top of the bed was flowing with an inconsistent velocity rather than smoothly, or if the beds came to a complete stop, steady state was not a valid assumption. In these instances, the data was thrown out, the reactor was re-charged and the run repeated. There may have been instances where these symptoms were not obvious enough to throw out a run, and this would have introduced error to the measurements. How much effect this had on the data is not clear, and thus it may account for the most significant source of error.

Discussion

The results gathered in the experiment showed rather close agreement with expectation. The slopes of the vb' vs vs' should all be close to unity if the two velocities are in fact equal. Further, the fitted intercepts were all fairly close to zero. Thus, at least for the case when the exit valve is closed the experimental results verified that neither column should have flow.

There is a deviation from the expected slope of unity for the angles between 50 and 60 degrees. It should be noted that the angles of 55 and 60 degrees were tested over a rather small range of percent valve openings, as can be seen in Figure 14 by the closeness of the data points. Had a wider range of velocities been tested, the slopes of these two lines may have been closer to one. However, the angle of 65 degrees also had a fairly limited range of velocities tested (though at higher magnitudes) and still showed a slope close to unity when fit to a line. Additionally, the angle of 50 degrees had a fairly wide range of velocities tested, though the slope of its linear fit is somewhat closer to one than the other outliers.

The assumptions inherent in the formulation of the insert velocities may also be the cause of the break in trend. For example, it was assumed that the bed density does not vary with axial reactor location so that a volume balance could be used between the velocity in the region measured and that near the insert. However, the bed is actually composed of a size distribution of particles from powder known as fines up to the 1.5 mm particles. This occurs because the particles break down during operation. The batches tested were mostly composed of the desired 1.5 mm particles, however there was a small quantity of small particle present. These smaller particles cause the constant volumetric flow rate assumption to lose validity. However, the

impact this has should not be significant as the presence of fines and other small particles was limited.

Another assumption made was that the velocity at the insert of the one inch column does not vary with radial or angular position. Because of the somewhat invasive nature of the insert, this may not be the case. The curvature of the top of the insert may cause friction to the flow in the three inch column, causing a difference in potential for the flow between the two columns. This may cause slight differences in the velocities of the two regions, located just below the region where friction is generated. However, it seems more likely that this effect would cause a break in trend for a region near the extremes of angles, not in the middle as is the case.

The criteria set for being able to correlate the velocity in an isokinetic device to that in the downcomer section which it is being used for was that the velocities of the two columns at the insert must be directly proportional. In theory the constant of proportionality should be one, or close to one, since the velocities at the insert are predicted to be equal. However, friction effects from the walls and the presence of a size distribution of solids may cause the constant to be larger or smaller. Regardless, the constant of proportionality can then be used to calculate the solids circulation rate in the CFB from measurements taken on the isokinetic device. From the experiments, it has been shown that the criteria for proportionality holds and most of the constants are very near one. Those that are not can still be used to correlate the two column velocities.

It can be concluded that the data collected during these experiments can be used to generate correlation factors and equations relating the solids velocity in an Iso-Kinetic Device to

the solids circulation rate in the downcomer section of a circulating fluidized bed. The relating equation would have the following form.

$$v'_b = m * v_s' + b \quad (6)$$

The constant m is the correlation factor generated from the slopes of the linear fits discussed previously. The constant b is the intercept also discussed earlier. Table 1 below displays the experimentally determined m and b for each angle tested.

Angle	m	b
30	0.91979	0.00787
35	0.87885	0.02707
40	0.85253	-0.0024
45	0.90454	0.00404
50	0.80095	-0.0143
55	0.57725	0.00642
60	0.4473	0.00597
65	1.05637	-0.1407
70	0.91733	0.00367
80	0.86878	0.02157

Table 1: The Correlation Constants for the Iso-Kinetic Device for Each Angle

Depending on how good of an approximation is required for the solids circulation rate; it may be possible to disregard the constant b. As has been discussed, this constant should be very close to zero, and for most of the angles tested it is.

Recommendations

A few recommendations for future research in this field will now be addressed. In order to more fully account for the effects of friction on the iso-kinetic devices reliability, it might be appropriate to use an insert fabricated out of the materials used in a commercial scale reactor.

The rest of the column could still be acrylic such that bed height measurements can be performed. This would allow the differences in particle wall interactions to be accounted for in the region where friction affects both columns. The angled inner wall of the insert is the most significant source of friction for the iso-kinetic device, and the top of the insert is for the downcomer. Being able to account for this in generating correlations would allow for more accurate results for use in a larger scale system.

Further, it is important to calculate specific correlation constants for use in an actual reactor as the nature of flow is very different, with the high temperature and the presence of upward directed gas flow.

Conclusion

Coal combustion through use of iron oxide particles as oxygen carriers in a chemical looping process is a novel means of meeting the recent demands on environmental sensitivity. The process is capable of inherently separating out the carbon dioxide produced through combustion in a pure stream such that carbon capture is more efficient. The process is also capable of producing hydrogen, in addition to producing high temperature/pressure gas streams for generating electricity using turbines. In the reactor portion of the process, the solids can be circulated through a loop using gas-solid fluidization for the portion that requires motion against gravity, and moving beds for the portion that can utilize gravity. The riser is the region where fluidization occurs; here the flow regime depends on solids circulation rate and inlet gas velocity. For this reason, it is important to be able to measure solids circulation rate. However, this is non-simple in systems where a reaction is taking place. The high temperature and pressure conditions within the reactor vessels make conventional flow measuring techniques particularly troublesome. For this reason, a device which does not require direct exposure of equipment to these conditions has been proposed. The iso-kinetic device involves a separate column with particles being inserted into the downcomer of a circulating fluidized bed. The solids flow rates in this separate column can be correlated to a solids circulation rate. The importance of entry angle for the insert of the device was tested in this research to determined correlation constants and to ensure all of the angles tested produced reliable correlation factors. The constants were calculated and it was verified that the solids velocity in the iso-kinetic device is directly proportional to the solids velocity in the downcomer for all angles tested. Thus, the iso-kinetic device is suitable for the purposes of solids circulation rate determination in this range.

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Appendix

Raw Data

	30 Degrees					35 Degrees				
	Time (s)	dHs (in)	dHb (in)	Vel,s (cm/s)	Vel,b (cm/s)	Time	dHs	dHb	Vel,s (cm/s)	Vel,b (cm/s)
Run # 1	53	14	5	0.6709434	0.23962264	66	14	6	0.53878788	0.23090909
Run #2	38	14	5	0.93578947	0.33421053	33	14	6	1.07757576	0.46181818
Run #3	98	14	5.25	0.36285714	0.13607143	40	14	6	0.889	0.381
Run #4	25	14	5	1.4224	0.508	21	14	5.75	1.69333333	0.69547619
		Average:	5.0625				Average:	5.9375		
		Relative Height Change:	0.361607				Relative Height Change:	0.424107		

40 Degrees					45 Degrees				
Time	dHs	dHb	Vel,s (cm/	Vel,b (cm	Time	dHs	dHb	Vel,s (cm/	Vel,b (cm
44	14	6.25	0.808182	0.360795	96	20	10.75	0.529167	0.284427
32	14	6.25	1.11125	0.496094	135	20	11	0.376296	0.206963
20	14	6.25	1.778	0.79375	30	20	10.7	1.693333	0.905933
58	14	6.2	0.613103	0.271517	251	18	9.75	0.182151	0.098665
	Average:	6.2375				Average:	10.55		
	Relative Height Change:	0.445536				Relative Height Change:	0.541026		

50 Degrees					55 Degrees				
Time	dHs	dHb	Vel,s (cm/s)	Vel,b (cm/s)	Time	dHs	dHb	Vel,s (cm/s)	Vel,b (cm/s)
162	17	8.5	0.26654321	0.1332716	145	19	8.2	0.33282759	0.14364138
64	18	8.6	0.714375	0.3413125	145	18	7.5	0.31531034	0.13137931
18	19	9.8	2.68111111	1.38288889	92	18.5	7.6	0.51076087	0.20982609
176	18.5	9	0.26698864	0.12988636	48	18	7.4	0.9525	0.39158333
152	18	8.85	0.30078947	0.14788816		Average:	7.675		
	Average:	8.95				Relative Height Change:	0.417687		
	Relative Height Change:	0.494475							

60 Degrees					65 Degrees				
Time	dHs	dHb	Vel,s (cm/s)	Vel,b (cm/s)	Time	dHs	dHb	Vel,s (cm/s)	Vel,b (cm/s)
179	21	7.5	0.29798883	0.10642458	35	14	10	1.016	0.72571429
71	21	7.1	0.75126761	0.254	34	14	10	1.04588235	0.74705882
161	16	5.5	0.25242236	0.08677019	39	14	9.8	0.91179487	0.63825641
90	20	7	0.56444444	0.19755556	37	14	9.8	0.96108108	0.67275676
	Average:	6.775				Average:	9.9		
	Relative Height Change:	0.347436				Relative Height Change:	0.707143		

70 Degrees					80 Degrees				
Time	dHs	dHb	Vel,s (cm/s)	Vel,b (cm/s)	Time	dHs	dHb	Vel,s (cm/s)	Vel,b (cm/s)
32	14	10.75	1.11125	0.85328125	40	14	11	0.889	0.6985
22	14	10.5	1.61636364	1.21227273	21	14	10.6	1.69333333	1.28209524
92	14	10.6	0.38652174	0.29265217	50	14	11	0.7112	0.5588
69	14	10.5	0.51536232	0.38652174	109	14	10.8	0.32623853	0.25166972
	Average:	10.5875				Average:	10.85		
	Relative velocity:	0.75625				Relative velocity:	0.775		

Calculated Data

	1" Pipe		3" PIPE	
angle	I. D.	W. Thck.	I. D.	T. A.
30	1.05	0.13	3.07	7.39
35	1.05	0.13	3.07	7.39
40	1.05	0.13	3.07	7.39
45	1.05	0.13	3.07	7.39
50	1.05	0.13	3.07	7.39
55	1.05	0.13	3.07	7.39
60	1.05	0.13	3.07	7.39
65	1.05	0.13	3.07	7.39
70	1.05	0.13	3.07	7.39
80	1.05	0.13	3.07	7.39

			1 inch		3 inch
Oval:		Insert:			
Dia Oval	Oval Area		Area:		Area:
2.099931	1.729219		1.729219		5.66
1.830706	1.507522		1.507522		5.88
1.633734	1.345323		1.345323		6.04
1.485283	1.223079		1.223079		6.17
1.371181	1.12912		1.12912		6.26
1.282487	1.056083		1.056083		6.33
1.213324	0.99913		0.99913		6.39
1.159732	0.954999		0.954999		6.43
1.119018	0.921472		0.921472		6.47
1.070129	0.881214		0.881214		6.51

Angle	Slope	Expected	Intercept	Abs	Expected
30	1.086988	1	-0.00847	0.008473	0
35	1.136373	1	-0.02998	0.029984	0
40	1.172961	1	0.00281	0.00281	0
45	1.105495	1	-0.00445	0.004446	0
50	1.248097	1	0.018049	0.018049	0
55	1.731508	1	-0.0109	0.010904	0
60	2.232059	1	-0.01269	0.01269	0
65	0.942474	1	0.136496	0.136496	0
70	1.089589	1	-0.00358	0.003583	0
80	1.150221	1	-0.02419	0.024189	0